

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:
Arjun G. Yodh et al. Confirmation No.: 7568
Application No.: 10/526,941 Group Art Unit: 1793
Filing Date: September 8, 2005 Examiner: Brittany M. Martinez
For: **Carbon Nanotubes: High Solids Dispersions and Nematic Gels Thereof**
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

DECLARATION PURSUANT TO 37 C.F.R. § 1.131

I, Mohammad F. Islam, hereby declare that:

1. I am an inventor of the invention described and claimed in U.S. Patent Application Number 10/526,941 (hereinafter referred to as "the 941 application"), filed September 8, 2005, in the United States Patent and Trademark Office.

2. I am aware that the pending claims of the 941 application have been rejected as being unpatentable over U.S. Patent Application Pub. No. 2003/0133865 ("Smalley"). It has been explained to me that Smalley was filed on July 2, 2002, but that it claims priority from four U.S. provisional applications (collectively referred to as "the Smalley provisionals"):

Serial No. 60/303,469, filed July 6, 2001;
Serial No. 60/303,470, filed July 6, 2001;
Serial No. 60/337,561, filed November 8, 2001; and
Serial No. 60/337,951, filed December 7, 2001.

3. It has been explained to me that none of the Smalley provisionals disclose the use of at least one surfactant comprising an aromatic group.

4. In accordance with CFR § 1.131, as an inventor of the subject matter of the pending claims, and without conceding the propriety of the rejections of the pending claims, I hereby declare that I invented the subject matter with the inclusion of at least one surfactant comprising an aromatic group prior to July 2, 2002. I further hereby declare that I worked diligently from a date prior to July 2, 2002, to the date of constructive reduction to practice, September 10, 2002, the priority date of the 941 application, in order to prepare the 941 application and patent the invention.

5. In support of the instant declaration, a copy of relevant pages of a laboratory notebook prepared during the development of the claimed invention is attached hereto (Attachment B), which was created prior to July 2, 2002. The date range is from May 22, 2002 through July 20, 2002, which provides evidence of conception of the invention prior to the effective date of the Smalley reference and evidence of due diligence from prior to said date to the filing date of the provisional application, September 10, 2002.

6. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information or belief are believed to be true; and further, that these statements were made with the knowledge that willful false statements and the like are punishable by fine or by imprisonment, or both, under § 1001 of Title 18 of the United States Code, and that such willful statements may jeopardize the validity of the application, any patent issuing thereupon, or any patent to which this verified statement is directed.

Date: 01/20/2010

Signature:

M.F. Islam

Mohammad F. Islam

Exhibit A

Chemical Engineering and Material Science and Engineering
Carnegie Mellon University
500 Forbes Avenue
Pittsburgh, PA 15213-3890
412.268.0593
FAX: 412.268.7586
mohammad@andrew.cmu.edu

EDUCATION

- 2000 Ph.D. Physics, Lehigh University, Bethlehem, PA 18015
1996 M.S. Physics, Lehigh University, Bethlehem, PA 18015
1994 B.S. Physics.

Work Experience

- | | |
|-----------------|---|
| WORK EXPERIENCE | |
| 2005-Present | Associate Professor, Chemical Engineering and Materials Science and Engineering
Carnegie Mellon University, Pittsburgh, PA 15213 |
| 2002-2005 | Postdoctoral Fellow, Department of Physics and Astronomy
University of Pennsylvania, Philadelphia, PA |

HONORS AND AWARDS

- HONORS AND AWARDS**

 - 2007 Alfred P. Sloan Research Fellow
 - 2007 National Science Foundation Career Award
 - 2006 American Chemical Society PRF Award
 - 1999 Sigma Xi
 - 1997 Hoechst Celanese Award for Excellence in Polymer Science

CURRENT RESEARCH INTERESTS

Novel Self-Assembly and Phase Transformations in Single- and Multi-Component Systems: Formation of diverse structures as well as phase transformations in multi-component systems using temperature sensitive colloidal particles

Utilizing Nanomaterials to Investigate Cellular Functions: Developing novel nanomaterial based vectors and investigating changes in cellular functions due to internalization of these vectors.

Carbon Nanotube Based Porous Materials for Energy Applications: Created ultra-light, highly porous materials with carbon nanotubes; Investigating use as electrodes and support for catalyst particles.

Dependence of Cell Functions on Substrate Properties: Developed polymeric hydrogels with tunable local stiffness and organization; Using materials to probe dependence of cellular functions on substrate stiffness and spatial organization

Exhibit B

05/22/02

Make 2 samples of 1% H₂PO₄ + 10X NaDDBS in water
each sample weighs 3g

add H₂PO₄ 0.03g 0.0321g 0.0324g

Make

Add	<u>NaDDBS</u>	2.002 g	{	19.96 %
	<u>Starch</u>			
	Total	10.03		

Add	H ₂ PO ₄	0.0321g	0.0324g
	NaDDBS	1.5221g	1.5103g
	water	1.4659g	1.4762g

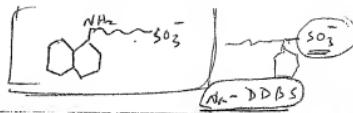
Sample #3

H₂PO₄ 0.0314g

NaDDBS 1.501g

water 1.480g

Dioxy Cholate



NT Project :

1. Scheme to separate & stabilize CNT without damaging it.

Tip Sonication (low f, high power) Both Batiksonication (high f)

- Advantage over pyrene functionalized CNT
(damages CNT & etc. destroys electronic property)

comment on Surfactant type for longer & better stability & separation

* Benzene a large surfactant best (NADDBS)

{ Literature is mostly on SDS
one paper ODA }

2. Fractionate CNT using either HPLC, SEC or GPC

↑
Can use
this

we use this

One paper on using GPC but do not elaborate or show careful results. (Duesberg's group)

- Can use this technique for other rod shaped particles

3. Characterization:

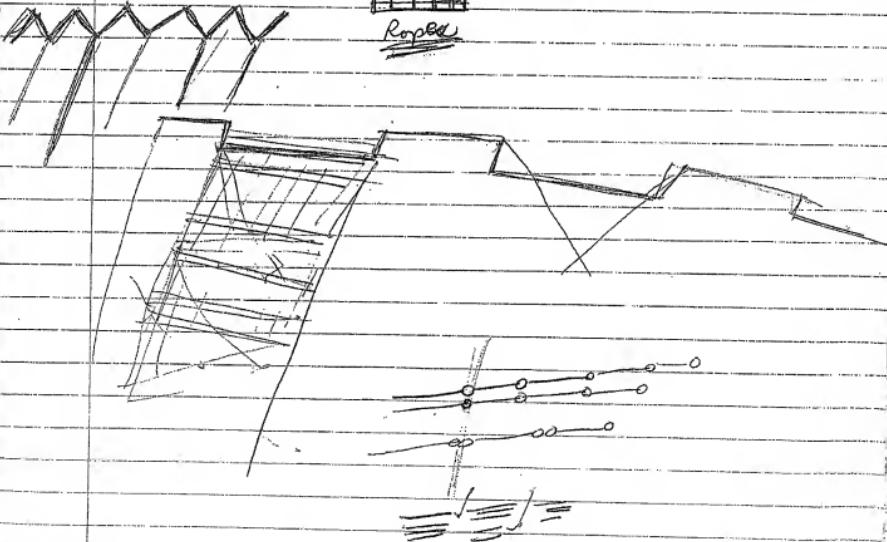
Use mainly LS ← large statistics

Cross check w/ a few portions w/
AFM

4. Impact: Stable, single CNT dispersions

Assembly, Controlled deposition ..

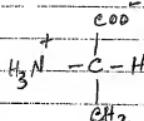
Roped



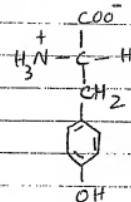
05/19/02

Bill Degrado

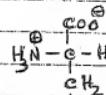
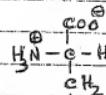
the more surfactants to try:



Phenyl alanine
(F)
Phe



Tyrosine



Tryptophan(W)
[Trp] ➤

or Trimer of F (F_3)

Also try Chaps & PEG

chiral proteins Bill Degrado JMB 2001

Butyl group, OH, benzene, amide group

Can we make chiral molecules to wrap around?
Use Hydrogen bonding.

06/18/02

Current Status of NT dispersion & characterization:

1% NT in NaDDBS

Bath sonic
dispersion

tipsonic
dispersion

electro-
phoresis

Run through
Sepharose 2B

AFM of
sample as is

electrophoresis
through 0.5%
Agarose
gel

AFM of
sample as is

Agarose
gel

sample
comes through
the gel.

Unsuccessful;
sample does
not stick
to surface

most did not
go through;
seems tip is not
a good technique
to disperse HPGs

unsuccessful;
sample does
not stick
to surface

into
small
segments

some of the
solutn gets
trapped into
the gel

modifies surface

Abandon this
method

when met
autolytic
enzymes

gel at
through 0.5%
filter (long elution
time)

saline

polyacrylate

successful;

center

They are
presumably
amorphous

Similar
as poly-L-L

Hipersolite to
surface after
dipping into soln
of HPG Co

most
of

gels
are
one
out
filter
every

surface quality

hipersolite to
surface after
dipping into soln
of HPG Co

filter

(Carbon,
bulky ball

& see if NT

next step
check the surface
quality & see
how NT works like

with

the
surface

sticks to the

next step
check the surface
quality & see
how NT works like

good
tech
wipes

collect - sample

multiple

next step
check the surface
quality & see
how NT works like

abandon

multiple

vials.

Try this
approach
more

next step

look it a few

of them to see
if how good is the
separation

07/01/2002

MT 10-4.000 Silane treated 10^{-4} chips 5um Scan in NADIR
(Too much surf)

MI 10-6.001 " " 10^{-6} " "
(Too much surf) start

MI 10-6.002 diff area, same chip

→ 10-4.003 10^{-4} chip rinsed in H₂O ~2 min

10-4.004 diff area, same chip

10-4.005 10^{-4} prepared by dunking in soin &
then rinsing with water, dried
with canned air

07/08/2002

Prepare 4g of 0.01% NT in NADDS from 1% NT in NADDS

1% NT soln ~~0.049~~ 0.044779 }
water 4.44349 } 0.01%

07/08/02

AFM on NAOBS stabilized LiPco

ddbs10-4.000

zoom on the surface, leave it on
for 10min, spin it at 3000 rpm
then add 2ml of water on the
sample is spinning.

→ no feature

ddbs10-4.001

same sample - diff location

ddbs10-4.002

dipped in soln, then for
5 min, then gently rinse
it in a water bath
- lots of NT

ddbs10-4.003

ZOOMED in some where
in the previous ~~the~~ picture

.004

different place on sample (SP)

.005

Zoom in of .004 (SP)

.006

new location (on (SP))

ddds10-4.007

12th cut of gel
physisis sample

cone $\approx 10^{-4}$ NT in NODBS

I dipped the chip
in sohn & then dried
& then rinsed in
water.

07/11/02

what I need to do:

conc

SDS

Tx100

No DDBS

Prepare 1% → dilute to 0.01%



~~Pre~~ sonicate donot sonicate;
just dilution

Prepare 0.01% sample ✓ ✓



Sonicate

I want to make 10% HiPCO soln.

HiPCO 0.2009g

9.96% Add Rest 20g NADDs to get 2.009g

Final wt 2.0461g

07/12/02

Make 20 wty TX-100 stock soln (50 grams)

TX-100 add 10 g

10.00 ~~8619~~

Rest add water till 50g

Total 50.0 ~~3274~~ 9

~~2~~
Make 49.9% TX-100 AdT soln w/ TX-100 (10%)

$$NT = 0.03979 \quad \left. \right\}$$

$$\left. \begin{array}{l} TX-100 \\ (20 \text{ wt\%}) \end{array} \right. = 2.00859 \quad \left. \begin{array}{l} \text{nr 1\%} \\ (0.984) \end{array} \right.$$

$$\text{water} = 1.9913$$

~~2~~
Also make 10^{-4} soln from 10^2 soln for ATM

SDS NT

$$(1.50/\text{m} = 0.045897) \quad \left. \begin{array}{l} \text{nr } 1 \times 10^{-4} \\ (6.93 \times 10^{-5}) \end{array} \right.$$

add water till = 4.60469g

NADDBS NT

$$\left. \begin{array}{l} 1.50/\text{m} = 0.04059 \\ \text{add water till} = 4.01145 \end{array} \right\} \quad \text{nr } 1 \times 10^{-4}$$

07/18/02

NT samples I have been looking at:

A

NADDBS coated

NT

10^{-4} dipped rinsed

Baked

ddbs dep. 0.00 < 5 μm

{ ddbs 10-40.40 }

- .051

4 μm scan

A1

Same

one A

bent not baked

2 μm scan

naddbsa1.026 }

- .037

{ ddbs 10-4.021

.026

5 μm
scan

discard
double tip

E1

1' NADDBS NT

Spun at 6000 rpm
while I pipetted

on NT salt

{ Naddbsel.000
before rigorous rinsing }

- .004

{ After rigorous rinsing }
naddbsel.005

- .015

same baked

naddbsel.040

bad

B2 NADDBSNT

1' 4000 rpm

Nadd. salt dropped

air ship iron

spinning

naddbsb2.000 } before

.002 } cleaning

naddbsb2.003 & after cleaning

Baked

naddbsb2.040 } same way
prepared

.042 } sample

overlapping conc. NT

E

NADDBS 1'

(sample @ month old)

Spins deposited

naddbsel.000 & before clean

.001 & after clean

.000 } conc. NT

NADDBS NT

1'

6000 rpm

spins deposited

2 M

{ Naddbsb.000 }
.041

image comparison

Baked

overlapping

Baked

naddbs.040 } same, NT

.042 } =

288.

70/307

158/307 ~ 60%
212/307 ~ 73%

E
 10^{-4} from 10^{-2} dipper

Naddbs 22.0003 before
baking

Naddbs 22.0407 after
.048 baking

F
 10^{-4} from 10^{-2} sample (1)
dipper

After baking

Naddbs 22.0406 } no tube
.041 }

no tube

Cut 12 new

12-2

ddbs 12 n. 000 { Long
. 005 } tubes

ddbs 12 - 2. 000 } rotubes
} _____

12th cut New-2

4th cut New1

ddBS 4N 1,000

↓
.009

Found single
tubes

c2

(dirty tube)

10^{-4} from 10^{-2}

dipped, rinsed

& baked

c3

10^{-4} from 10^{-2} dipped, rinsed
baked

a lot of tubes

f2

10^{-4} from 10^{-2}
(sample ①)

dipped, rinsed
baked

Noddhs F2,000

↓
.009

G1

10^{-3} from 10^{-2}

dmsoda, rinsed

& baked

c4

10^{-4} from 10^{-2} dipped,
baked

a lot of tubes but

chip is dirty.

H1

H2

Bare surface
as
silane
H1

treated, rinsed but
baked

not
rinsed

baked

07/20/02

Prepare high conc. of Laser Tuber

Take 20 mg of 5×10^{-3} wt tubes \rightarrow in a glass bottle
& slowly evaporate water to increase conc.

$$\text{Empty bottle w/o cap} = 13.9254 \text{ g}$$

$$\underline{\text{w/tube w/o cap}} \quad 28.92 \cancel{7.9} \text{ g}$$

$$\therefore \underline{\text{tube wt}} = \underline{15.002 \text{ g at } 0.5^\circ \text{ wt}}$$

Slowly evaporate at 44°C for several days

$$\text{After some solvent evaporation} \quad \underline{\text{w/tube w/o Cap}} = 17.4744$$

$$\therefore \text{tube soln. wt} = 3.549$$

New conc -

$$\text{New conc} = 2.113 \times 10^6 \cancel{\text{g}} \text{ at } ^2 = 2.113 \cancel{7.5} \text{ g wt}$$

Prepare 0.1% NaDBS-NT soln to sonicate

07/24/02

$$1\% \text{ NaDBS-NT} = 0.4038 \text{ g}$$
$$\text{Add water till} = 4.0127 \text{ g}$$
$$\left. \begin{array}{l} \\ \\ \end{array} \right\} \approx 0.1\%$$

Prepare 0.1% SDS-NT soln

$$1\% \text{ SDS-NT} = 0.409 \text{ g}$$
$$\text{Add water till} = 4.0193 \text{ g}$$
$$\left. \begin{array}{l} \\ \\ \end{array} \right\}$$